SUSTAINABLE BIOCHAR ASSISTED NANOCOMPOSITES FOR EFFICIENT REMEDIATION OF NOXIOUS CONTAMINANTS

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Lakhwinder Singh

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Under the Supervision of

Dr. Harpreet Kaur

DEPARTMENT OF CHEMISTRY

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CERTIFICATE

This is certified that synopsis of Lakhwinder Singh, Ph.D. Chemistry student of School of Sciences (SOS) has been finalised in consultation with research guiding committee. The student has successfully delivered his synopsis seminar entitled "Sustainable biochar assisted nanocomposites for efficient remediation of noxious contaminants" on date 17 September 2024 in accordance with the academic regulation. The suggestions given by the members have been duly incorporated.

Dr. Harpreet Kaur

Major Research Guide

Members of Research Guiding Committee

17.09.2024

Dr. Kamal Kishore Thakur
Associate Professor

External Member
Department of Chemistry
Chandigarh University

Dr. Gaurav Thakur
Assistant Professor

Internal Member
Department of Microbiology
BUEST

Dr. Arush Sharma
Assistant Professor

Internal Member
Department of Chemistry
BUEST

COUNTERSIGNED BY

Dr. Nidhi Gupta

Associate Dean, School of Sciences

Prof. (Dr.) Ravinesh Mishra

Dean R&D

APPROVED BY

Dean Academics Affairs

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1. INTRODUCTION

Our environment can be defined as the sum of total living and non-living things around us and how they affect the human life. There are living things or biotic things like plants, animals, forest, birds and non-living things or abiotic things like forest, rocks, sunlight, water, air etc. But now a day our environment is deteriorating day by day. The releases of harmful material from industries cause bad effect to the environment and human health also. Heavy metals and minerals in the waste water are the main cause of environmental pollution. There are very common pollutants present in the waste water like phenols, herbicides, pesticides, detergent, dyes, and also the aromatic compounds that cause the environmental pollution. Exploring the depths of water pollution aims to contribute to the ongoing dialogue on the environmental conservation by providing a comprehensive understanding of water pollution intricate challenges and potential solutions. Through this research the hope is to inspire informed decision making, policy formulation and grassroots initiatives that will collectively be related to cleaner and healthier water bodies for present and future generations. The contamination of water bodies can occur due to various types of pollutants, originating from both natural and human related activities. These pollutants can have detrimental effect on aquatic ecosystem, human health and the environment. There are many pollutants which cause severe effects on human health when released into water. These pollutants are categorized into main categories i.e. organic pollutants and inorganic pollutants. Nanotechnology involves manipulating matter at the atomic or molecular scale, typically between 1 and 100 nanometres. Nanoparticles (NP's) exhibit unique properties, such as high surface area-to-volume ratios and quantum effects, making them highly effective for various applications, including environmental remediation (Nair, R., *et al., 2010*).

Biochar is produced through the pyrolysis of biomass in an oxygen limited environment, resulting in a stable form of carbon with high surface area and porosity. Its properties, including surface chemistry and adsorption capacity, make it an excellent material for environmental applications (Lehmann, J., *et al., 2015*). Biochar has been shown to improve soil fertility, sequester carbon, and mitigate greenhouse gas emissions. Its adsorption properties make it useful for removing contaminants from water and soil (Atkinson, C., *et al., 2010*). Nanoparticles are materials with dimensions in the nanometre range that exhibit unique chemical and physical properties due to their small size. Common types include metal oxides (e.g., Ti, Fe), metal nanoparticles (e.g., Au, Ag), and composite nanoparticles (Nair, R.., *et al., 2010*). Nanoparticles can be synthesized using various methods, including physical

vapour deposition, chemical vapour deposition, sol-gel processes, and hydrothermal synthesis. The choice of method influences the size, shape, and properties of the nanoparticles (Wang, X., *et al., 2015*). The integration of nanoparticles with biochar aims to enhance the adsorption capacity, catalytic activity, and stability of biochar. This combination leverages the advantages of both materials to improve environmental remediation technologies (Wang, J., *et al., 2018*). Metal oxides such as titanium dioxide and iron oxides are commonly used in biochar composites due to their photocatalytic property. Titanium dioxide biochar composites are effective in photocatalytic degradation of organic pollutants under UV light (Zhang, X., *et al., 2017*). Metal nanoparticles like silver (Ag) and gold (Au) are incorporated into biochar to enhance antimicrobial properties and pollutant reduction. For example, Ag-biochar composites exhibit high antibacterial activity and can be used in water disinfection (Liu, Y., *et al., 2016*). Magnetic nanoparticles combined with biochar facilitate the easy separation of the composite material from water using an external magnetic field. Like, iron oxide biochar composites are used for the removal of heavy metals and organic pollutants, with the added benefit of magnetic recovery (Zhang, X., *et al., 2019*).

The Co-precipitation involves the simultaneous precipitation of nanoparticles and biochar from a solution. This method is often used for creating metal oxide-biochar composites (Han, L., *et al., 2014*). Hydrothermal synthesis uses high-pressure and high-temperature conditions to produce nanoparticles that are then incorporated into biochar (Li, J., *et al., 2018*). The sol-gel process involves the formation of a gel that contains nanoparticles, which is then mixed with biochar (Yang, X., *et al., 2017*). Nanoparticle biochar composites are highly effective in removing heavy metals such as lead (Pb), cadmium (Cd), and arsenic (As) from contaminated water due to their high adsorption capacities and catalytic properties (Wang, Q., *et al., 2020*). The photocatalytic properties of metal oxide-biochar composites facilitate the degradation of organic pollutants like dyes and pharmaceuticals (Kim, H., *et al., 2018*).

In soil applications, nanoparticle-biochar composites can enhance contaminant removal and improve soil health by providing additional nutrients and improving soil structure (Xie, Y., *et al., 2020*). Assessing the potential environmental impacts, including the leaching of nanoparticles and their effects on ecosystems, is essential for ensuring safe use (Xia, X., *et al., 2020*). The photocatalytic and catalytic properties of these composites are utilized for the degradation of organic pollutants such as dyes and pharmaceuticals (Kim, H., *et al., 2019*).

Nanoparticle-biochar composites are applied in water and soil treatment processes to enhance pollutant removal efficiency and improve overall environmental quality (Xie, Y., *et al., 2020*). Ensuring the stability and minimizing the leaching of nanoparticles from biochar composites is critical for their long-term effectiveness and environmental safety (Kumar, P., *et al., 2019*). The cost of synthesizing and utilizing nanoparticle-biochar composites needs to be evaluated to ensure that they are economically viable for large-scale applications (Zhang, S., *et al., 2021*).

A study by Venkatesh et al. (2016) optimized co-precipitation conditions to control the size of silver nanoparticles. By adjusting parameters like temperature, concentration, and pH, they achieved nanoparticles with uniform size and morphology (Venkatesh, V., *et al., 2016*). Siddiqui et al. (2017) compared various reducing agents like sodium citrate, ascorbic acid, and hydrazine in the co-precipitation synthesis of silver nanoparticles, evaluating their effectiveness in controlling particle size and morphology (Siddiqui, M. R., *et al., 2017*). Economic feasibility is a key factor in scaling up nanoparticle production. The study by Wang et al. (2020) evaluated the cost-effectiveness of the co-precipitation method compared to other synthesis techniques, providing insights into its economic viability for large-scale applications (Wang, J., *et al., 2020*).

Each of these technologies in activated carbon, biochar, and nanoparticles represents a significant advancement in water treatment capabilities. Their effectiveness, however, varies depending on the specific contaminants, local conditions, and treatment goals. Integrating these technologies with conventional methods can enhance overall water quality and contribute to sustainable environmental management. Continued research and development are essential for optimizing these technologies and ensuring their safe and efficient application in diverse water treatment contexts.

2. LITERATURE REVIEW

Wastewater treatment plays a crucial role in environmental sustainability by eliminating contaminants from water before it enters the water cycle. Effluents from various sources, such as industrial processes and domestic sewage, can significantly impact ecosystems if not properly treated. One notable contaminant often found in wastewater is dyes, which are aromatic organic compounds extensively used in industries like textiles, paper, and cosmetics for colouring substrates. The presence of dyes poses challenges due to their persistence and potential harm to aquatic life and human health.

Activated carbon (AC) emerges as a pivotal tool in wastewater treatment, particularly for removing dissolved organics like dyes. AC effectiveness lies in its highly porous structure, which provides a large surface area for adsorption. The production of AC involves processes like physical and chemical activation. Physical activation typically involves heating carbonaceous materials like biomass in the presence of an oxidizing gas, while chemical activation employs dehydrating agents such as $ZnCl_2$ or KOH to enhance pore formation and surface area. These methods not only remove tarry substances that could block pores but also increase the adsorptive capacity of the carbon.

A Green synthesis approach for producing silver nanoparticles using Beta vulgaris peel extract. This method involves utilizing the organic functional groups present in the extract to chemically reduce silver ions, forming nanoparticles. The research investigates how different process conditions influence the size distribution and surface structure of these nanoparticles. It identifies that controlled conditions, including induced intraparticle ripening, contribute to the formation of anisotropic silver nanoparticles embedded within the extract's matrix. The nanoparticles exhibit distinctive optical properties, with plasmon absorption and resonance scattering suitable for both small (below 25 nm) and larger (above 75 nm) sizes, indicating a narrow size distribution (Mehdizadeh, S., *et al., 2019*).

A green synthesis method for Ag-CMPB using Citrus maxima peel extract, emphasizing cost-effectiveness and eco-friendliness. Characterization via XRD, HRTEM, BET, FTIR, EDX, and PZC revealed FCC-structured silver nanoparticles (3-10 nm) within a biochar matrix. CMP extract components like flavonoids and phenolic acids acted as efficient reducing agents. This approach offers sustainable nanomaterials for environmental and biomedical applications (Tran, N.D.N., *et al., 2022*).

Magnesium nanoparticles are known for their high reactivity and potential in various applications including hydrogen storage and environmental remediation. They can be synthesized via methods such as chemical vapor deposition and ball milling (Wu, C., *et al., 2012*). Magnesium nanoparticles have been explored for their use in water treatment and soil remediation due to their reactivity with contaminants (Li, X., *et al., 2015*).

Research into the safety and potential toxicological effects of magnesium nanoparticles is ongoing, focusing on their environmental impact and interaction with biological systems (Kim, K., *et al., 2013*). Magnesium nanoparticles are incorporated into biochar using techniques such as co-precipitation and mechanochemical synthesis. Like, Magnesium nanoparticles can be integrated into biochar through co-precipitation from magnesium salts, creating composites suitable for contaminant removal (Zhang, X., *et al., 2017*).

Research by Singh et al. (2021) explored the use of magnesium nanoparticles in catalytic applications, demonstrating their effectiveness in various catalytic reactions, including hydrogen storage and organic transformations (Singh, A., *et al., 2021*). The potential of magnesium nanoparticles in biomedical applications, such as drug delivery and imaging, was investigated by Ali et al. (2022). The study highlighted the biocompatibility and efficacy of Mg NP's in medical applications (Ali, M., *et al., 2022*). Research by Zhao et al. (2021) assessed the environmental impact of using co-precipitation for magnesium nanoparticle synthesis, including potential hazards and strategies for minimizing environmental risks (Zhao, L., *et al., 2021*).

The combination of silver and magnesium nanoparticles with biochar aims to leverage the unique properties of each component, enhancing the composite's effectiveness for environmental remediation. Silver nanoparticles provide antimicrobial properties, while magnesium nanoparticles offer high reactivity for contaminant removal (Wang, Y., *et al., 2018*). Techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD) are used to characterize the structure and morphology of silver and magnesium nanoparticle-biochar composites (Kumar, P., *et al., 2017*). Surface area, porosity, and surface chemistry of the composites are analyzed using methods like BET surface area analysis and Fourier-transform infrared spectroscopy (FTIR) (Li, J., *et al., 2017*).

In soil applications, nanoparticle-biochar composites can enhance the removal of contaminants and improve soil health by adding valuable nutrients (Xie, Y., *et al., 2020*).

Ensuring the stability and longevity of silver and magnesium nanoparticle-biochar composites under environmental conditions is crucial. Research should focus on enhancing the durability of these materials (Kumar, P., *et al., 2019*). The cost of synthesizing and using nanoparticle-biochar composites can be high. Future research should aim to develop cost-effective synthesis methods and assess the economic viability of these technologies (Zhang, S., *et al., 2021*). The process generates a carbon rich material with high surface area and porosity, which is beneficial for adsorption applications (Zhang, X., *et al., 2018*).

Beetroot biochar has been studied for its ability to adsorb various contaminants from water and soil. Its effectiveness is attributed to its porous structure and high surface area (Zhang, Y., *et al., 2020*). Silver nanoparticles exhibit unique antimicrobial properties and high surface reactivity. They are typically synthesized using methods such as chemical reduction, biological synthesis, and physical techniques (Rai, M., *et al., 2009*). Silver nanoparticles are typically integrated into beetroot biochar using chemical reduction methods. This process involves reducing silver ions to metallic silver and embedding them onto the biochar surface (Li, H., *et al., 2015*). Magnesium nanoparticles are often synthesized and then incorporated into beetroot biochar via co-precipitation or mechanical methods. These methods ensure that magnesium nanoparticles are uniformly distributed within the biochar matrix (Zhang, X., *et al., 2017*). The surface area, porosity, and chemical functionality of the composites are analyzed using methods like Brunauer-Emmett-Teller (BET) surface area analysis and Fourier-transform infrared spectroscopy (FTIR) (Li, J., *et al., 2018*).

Nickel-doped cobalt ferrites are a class of spinel ferrites that have garnered attention for their magnetic, catalytic, and electronic properties. Doping cobalt ferrites with nickel can enhance these properties, making them suitable for various applications including magnetic resonance imaging (MRI), catalysts in industrial processes, and energy storage devices. This review discusses recent advancements in the synthesis of nickel-doped cobalt ferrites using nanotechnology techniques, emphasizing different preparation methods, characterization techniques, and applications. The co-precipitation method involves the simultaneous precipitation of nickel and cobalt ferrite components from aqueous solutions, followed by calcination to form the ferrite phase. This method is popular due to its simplicity and scalability (Patel, P., *et al., 2016*). Research by Sharma et al. (2017) focused on optimizing co-precipitation parameters such as pH, temperature, and metal ion concentration to enhance the properties of Ni-Co-Ferrite nanoparticles. The study also employed X-ray diffraction (XRD) and scanning electron microscopy (SEM) for characterization (Sharma, V., *et al.,*

2017). The sol-gel method involves the transition of a colloidal solution (sol) into a gel-like network that contains the metal ions. This method allows for precise control over the composition and morphology of the nanoparticles (Gupta, A., *et al., 2015*). The study by Mondal et al. (2018) explored the influence of different sol-gel processing parameters, such as aging time and calcination temperature, on the structural and magnetic properties of Ni-$CoFe_2O_4$ nanoparticles (Mondal, P., *et al., 2018*). The hydrothermal method uses high-pressure and high temperature conditions in an aqueous solution to synthesize nanoparticles. This method can produce high quality nanoparticles with controlled size and morphology (Huang, J., *et al., 2019*).

A study by Zhang et al. (2020) investigated the effect of hydrothermal synthesis parameters on the particle size and morphology of Ni-Co-Ferrites nanoparticles, highlighting the potential for tailoring magnetic properties (Zhang, X., *et al., 2020*). The solid state reaction method involves mixing metal oxides or hydroxides and heating them to high temperatures to form ferrite nanoparticles. This method is known for producing nanoparticles with high purity (Lee, K., *et al., 2021*). Research by Kumar et al. (2022) examined how varying the concentration of nickel in cobalt ferrites affects the structural and magnetic properties of the nanoparticles synthesized by solid state reactions (Kumar, R., *et al., 2022*).

VSM is employed to assess the magnetic properties of Ni-Co-Ferrites nanoparticles, including saturation magnetization and coercivity. The study by Li et al. (2018) explored the magnetic behavior of these nanoparticles in detail (Li, H., *et al., 2018*). Nickel-doped cobalt ferrites have shown potential as MRI contrast agents due to their superparamagnetic properties. The research by Chen et al. (2020) demonstrated the effectiveness of these nanoparticles in enhancing MRI imaging (Chen, Y., *et al., 2020*). Ni-Co-Ferrites nanoparticles have been investigated for catalytic applications, including environmental remediation and industrial reactions. The study by Patel et al. (2021) highlighted their efficacy as catalysts for the degradation of organic pollutants (Patel, S., *et al., 2021*).

The integration of nanoparticles with biochar has emerged as a significant area of research due to its potential applications in environmental remediation, catalysis, and energy storage. Silver, magnesium, and nickel-doped cobalt ferrites are particularly notable for their magnetic properties, catalytic activities, and antimicrobial effects. This review focuses on recent advancements in the synthesis of these nanoparticle composites with biochar, exploring various methods, characterization techniques, and applications.

Lanthanum nanoparticles are known for their catalytic properties and potential applications in environmental remediation. They are typically synthesized via chemical reduction, sol-gel processes, or hydrothermal methods (Zhang, H., *et al., 2016*).

Bismuth nanoparticles exhibit excellent photocatalytic activity and antimicrobial properties. Methods for their synthesis include chemical reduction and thermal decomposition (Chen, D., *et al., 2018*). Zinc nanoparticles are widely used for their antimicrobial properties and as catalysts in various chemical reactions. They are commonly synthesized via methods such as chemical vapor deposition and sol-gel processes (Xie, Y., *et al., 2014*). To enhance its adsorption capacity, biochar can be chemically or physically modified. Functionalization with various chemical groups improves its interaction with pollutants (Zhang, Y., *et al., 2020*).

Lanthanum nanoparticles are integrated into biochar using methods like impregnation, co-precipitation, or hydrothermal synthesis. These methods ensure uniform distribution and effective interaction between La NP's and biochar (Liu, Y., *et al., 2020*). Bismuth nanoparticles are incorporated into biochar matrices using techniques such as sol-gel processes and chemical reduction, which help in stabilizing Bi NP's on biochar surfaces (Chen, D., *et al., 2019*). Characterization of nanoparticle-biochar composites involves various techniques to assess their structural, morphological, and chemical properties, including SEM, TEM, XRD, and BET surface area analysis (Kumar, P., *et al., 2017*). Surface properties like porosity, surface area, and functional groups are analyzed using techniques such as Brunauer-Emmett-Teller (BET) analysis and Fourier-transform infrared spectroscopy (FTIR) (Li, J., *et al., 2018*).

Lanthanum, bismuth, and zinc nanoparticle-biochar composites have shown effectiveness in removing heavy metals like lead, cadmium, and arsenic from contaminated water (Zhang, Y., *et al., 2021*). The photocatalytic and catalytic properties of these composites are utilized for the degradation of organic pollutants such as dyes and pharmaceuticals (Kim, H., *et al., 2019*). Lead tree (Leucaena leucocephala) is a fast-growing, nitrogen fixing legume with high biomass production, making it an excellent feedstock for biochar production. Its biochar has shown promising properties for environmental applications, such as pollutant removal and soil improvement (Nair, R., *et al., 2010*). A study by Mousavi, S.M., et al. (2020) investigated the effects of different pyrolysis temperatures and heating rates on the yield and properties of Leucaena leucocephala leaf biochar. The research highlighted the optimal conditions for achieving desired biochar characteristics (Mousavi, S.M., *et al., 2020*).

Research by Chen, X., et al. (2019) explored the properties of biochar produced from Leucaena leucocephala leaves using hydrothermal carbonization and its potential applications in soil amendment and wastewater treatment (Chen, X., *et al., 2019*).

A comparative study by Rashid, M.A., et al. (2022) examined the properties of Leucaena leucocephala leaf biochar produced via gasification versus pyrolysis, highlighting differences in surface area, porosity, and chemical composition (Rashid, M.A., *et al., 2022*). Microwave-assisted pyrolysis uses microwave energy to heat biomass rapidly and uniformly. This method can improve the efficiency of biochar production and influence its properties (Sun, X., *et al., 2019*). The study by Li, X., et al. (2020) investigated the efficiency of microwave-assisted pyrolysis for producing Leucaena leucocephala leaf biochar and its impact on the biochar's physical and chemical properties (Li, X., *et al., 2020*). BET surface area analysis provides information on the surface area and porosity of biochar. Research by Khan, M.N., et al. (2018) used BET analysis to characterize the surface properties of Leucaena leucocephala leaf biochar (Khan, M.N., *et al., 2018*).

Lanthanum nanoparticles are known for their catalytic and adsorption properties. They are synthesized through methods such as chemical reduction, sol-gel processes, and hydrothermal synthesis. These nanoparticles are effective in removing contaminants from water due to their high surface area and reactivity (Zhang, H., *et al., 2016*). Bismuth nanoparticles are valued for their photocatalytic and antimicrobial properties. They are typically synthesized using techniques such as chemical reduction and thermal decomposition. Bi NP's are effective in degrading organic pollutants and pathogens (Chen, D., *et al., 2018*). Zinc nanoparticles are used for their antimicrobial properties and catalytic activities. They are commonly synthesized via chemical vapor deposition, sol-gel processes, and other methods. Zn NP's are effective in environmental remediation, including the removal of heavy metals and organic pollutants (Xie, Y., *et al., 2014*).

Biochar derived from lead tree (Leucaena leucocephala) is produced through pyrolysis, which involves heating the biomass in the absence of oxygen. The resulting biochar exhibits high surface area and adsorption capacity, making it a suitable support material for nanoparticles (Zhang, X., *et al., 2018*). A study by Khan et al. (2020) utilized techniques such as Brunauer-Emmett-Teller (BET) surface area analysis and Fourier-transform infrared spectroscopy (FTIR) to investigate the surface properties and functional groups of magnesium nanoparticles (Khan, M. N., *et al., 2020*). Research by Kumar et al. (2017) focused on

optimizing the synthesis parameters for lanthanum nanoparticles. The study examined the effects of varying pH, reaction time, and reagent concentration on the properties of the nanoparticles (Kumar, V., *et al., 2017*).

The study by Patel et al. (2018) investigated the use of stabilizers such as polyvinyl alcohol (PVA) and citric acid in the co-precipitation synthesis of lanthanum nanoparticles. The research highlighted how stabilizers influence the size, distribution, and stability of the nanoparticles (Patel, R., *et al., 2018*). A study by Gupta et al. (2019) explored the effect of various additives, including reducing agents and complexing agents, on the synthesis of lanthanum nanoparticles. The study aimed to improve the yield and quality of the nanoparticles (Gupta, N., *et al., 2019*).

The co-precipitation techniques are favored for its low cost and ease of scalability. However, controlling particle size and preventing agglomeration can be challenging. The technique also requires precise optimization of synthesis conditions to produce high quality nanoparticles (Liu, X., *et al., 2015*). One of the foundational studies by Singh et al. (2014) investigated the synthesis of zinc nanoparticles using co-precipitation with sodium hydroxide as the precipitating agent. The study explored the effects of reaction parameters on the size and morphology of Zn NP's (Singh, P., *et al., 2014*).

The synthesis of lanthanum, bismuth, and zinc nanoparticle composites with biochar derived from Leucaena leucocephalare presents a significant advancement in materials science, particularly for environmental and catalytic applications. Various synthesis methods, including co-precipitation, sol-gel, and hydrothermal techniques, offer distinct advantages in terms of particle size, crystallinity, and functional properties. Characterization techniques such as XRD, SEM, FTIR, and TEM provide crucial insights into the structural and functional attributes of these composites. The applications of these materials in environmental remediation and catalysis underscore their potential for addressing contemporary challenges in pollution control and sustainable development.

3. RESEARCH GAP

Recent advancements in wastewater treatment have undoubtedly been propelled by the utilization of advanced materials such as nanomaterials and modified adsorbents. These materials have shown promising capabilities in enhancing adsorption capacities and selectivity for a diverse array of pollutants. Despite these strides, significant research gaps persist that necessitate further exploration and development. One critical gap lies in the quest for new, sustainable, and cost-effective adsorbent materials that not only surpass current standards but also exhibit improved efficiency in pollutant removal across a broader spectrum. Moreover, while the application of advanced materials has been promising, understanding the intricate interplay of reactor design, flow dynamics, and mass transfer limitations remains rudimentary. Comprehensive investigations into these factors are imperative to optimize the integration of adsorption technologies into existing wastewater treatment infrastructure effectively. By bridging these gaps, researchers can unlock the full potential of adsorption processes, enabling sustainable and economically viable solutions for pollutant removal on a larger scale. This holistic approach is essential for advancing wastewater treatment technologies towards greater efficiency, reliability, and environmental sustainability in the face of evolving global challenges. The research on waste water treatment has made a significant progress in recent years, particularly with the applications of advanced materials like nanomaterials and modified adsorbent. Despite the progress made there is a need to explore and develop new, sustainable and cost-effective adsorbent materials with improved adsorption capacities and selectivity for wider range of pollutants. Factors such as reactor design, flow dynamics and mass transfer limitations remain poorly understood and need comprehensive investigation. Bridging this gap is essential to ensure the seamless integration of adsorption technology into existing waste water treatment infrastructure maximizing their potential for sustainable and cost-effective pollutant removal on a broader scale.

Translating laboratory scale success to real world applications is a significant challenge. There is limited research on the scalability of activated biochar supported nano-hetero assemblies and their performance in large scale water treatment systems. Issues such as material cost, production methods, and system integration require further investigation. While small scale studies have demonstrated the effectiveness of these materials, scaling up involves addressing issues related to the uniform distribution of nanoparticles on biochar, ensuring reproducibility, and managing production costs. Research has shown that while

biochar can be produced economically from agricultural residues, the cost of incorporating nanoparticles and ensuring their stability at scale remains a challenge (Lehmann, J., *et al., 2015*). Research has highlighted the risk of nanoparticle release from water treatment systems, which could lead to unintended ecological consequences. For instance, studies have reported the potential for silver nanoparticles, commonly used in water treatment, to exhibit antimicrobial properties that might affect non-target microorganisms in aquatic environments (Keller, A. A., *et al., 2015*).

While the regeneration and reusability of activated biochar-supported nano-hetero assemblies are crucial for their cost-effectiveness and sustainability, research in this area is limited. There is a need for studies that explore the methods for regenerating these materials without significant loss of performance. Some studies have investigated the regeneration of biochar materials, but there is limited research on the regeneration of nano-hetero assemblies supported by biochar. For example, while magnetic biochar composites can be regenerated using magnetic separation, the regeneration of biochar supported photocatalytic or catalytic nanoparticles requires further exploration (Chen, H., *et al., 2015*). Research is needed to assess how these materials perform under real world conditions where water may contain a mixture of pollutants and interfering substances Laboratory studies often use simplified water matrices, which may not accurately reflect the conditions in natural or industrial wastewater. For instance, the presence of competing ions or organic matter can affect the efficiency of contaminant removal by biochar supported nano-hetero assemblies (Mohan, D., *et al., 2015*).

Regulatory guidelines for nonmaterial in environmental applications are still evolving, and there is a need for comprehensive standards that address the safety, efficacy, and environmental impact of nano-hetero assemblies used in water treatment (Sadaf, S., *et al., 2015*). While the effectiveness of these materials has been demonstrated in laboratory settings, a comprehensive cost-benefit analysis that considers factors such as material production, operational costs, and maintenance is necessary to evaluate their viability for practical use (Gao, H., *et al., 2015*).

Research is needed to assess how these materials maintain their effectiveness over extended periods. Studies often focus on short-term performance evaluations, while long-term stability under real-world conditions remains less explored. For example, the impact of operational conditions such as flow rates, temperature variations, and pH changes on the stability and efficiency of these materials needs further investigation (Xie, Y., *et al., 2015*).

4. RESEARCH OBJECTIVES

Based on aforementioned discussion, current study deal with the following objectives:

1. To prepare activated biochar from *Beta vulgaris L. (Beetroot)* and *Leucaena leucocephala (Leadtrees)* plant waste material.

2. To synthesize the biochar supported Ag-Mg nanocomposite encapsulated Nickel-cobalt-ferrites.

3. To synthesize biochar supported lanthanum (Ln) doped Bismuth (Bi) - Zinc (Zn) nanocomposite using leaf extract by green synthesis method.

4.To characterize the prepared sample with various characterization techniques such as field emission scanning electron microscopy (FESEM), high resolution transmission electron microscopy (HRTEM), Fourier transform infrared microscopy (FTIR), X- ray diffraction (XRD), Bruner-Emmett-Teller (BET), energy Dispersive X-ray (EDX), vibrating-sample magnetometer (VSM) and UV- visible spectra etc.

5. To study the photocatalytic and adsorption remediation effect of prepared nanocomposites on emerging noxious contaminants.

5. RESEARCH METHODOLOGY

5.1. Chemicals Required

Chemicals required such as Silver nitrate ($AgNO_3$), Cobalt chloride ($CoCl_2$), Nickel sulfate ($NiSO_4$), Magnesium chloride ($MgCl_2$), Ferrous chloride ($FeCl_2$), sodium hydroxide (NaOH).

5.2. Synthesis of Activated Biochar (AB)

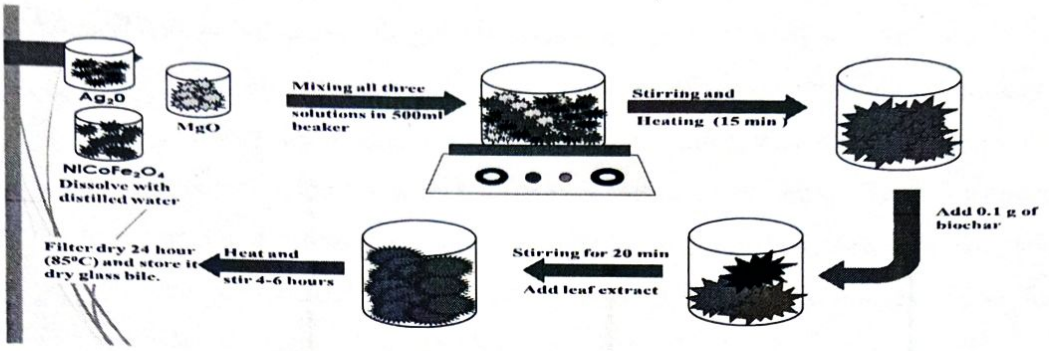
To prepare activated biochar from *Beta vulgaris L. (Beetroot)* leaf plant waste material was obtained from local vegetable market located in HMT city, Panchkula (Haryana) and *Leucaena leucocephala (Leadtrees)* plant waste material was obtained from the leadtrees in the forest area of Chandimandir city, Panchkula (Haryana). The selected materials were washed with distilled water to remove the contaminants, then sliced into small pieces and sieved to achieve a uniform particles size. The materials were then divided into two parts, one for physical activation and the other for chemical activation. In the first part, the material is dried in muffle furnace and refluxed for 8-9 hours. The resulting material was then withdrawn, filtered, and continuously washed with distilled water until the pH reached to 7.0 or neutral.

Scheme 1: Preparation of activated biochar from Beta vulgaris L. (Beetroot) plant waste material.

5.3. Green Synthesis of biochar supported Ag-Mg nanocomposite encapsulated Nickel-cobalt-ferrites.

To prepare a biochar supported Ag-Mg nanocomposite encapsulated in Nickel-Cobalt ferrites, dissolve silver oxide, magnesium oxide, and nickel-cobalt ferrite nanoparticles separately in 25 mL of distilled water, stirring each solution for 15 minutes. After preparation, all three solutions were mixed with constant stirring for an additional 15 minutes, then pour the mixture into a 500 mL beaker. Add 0.1 g of biochar (beetroot) to the solution, continuing to stir for 20 minutes, followed by the addition of 10 mL of leaf extract to provide a surface for green synthesis of the

reaction. Heat the solution while stirring for 4-6 hours, then filtered it and washed it several times with distilled water to remove impurities. After filtration, collect the nanocomposite and dried it for 24 hours in an oven at 85°C and , then stored it in a dry glass vial.


Ag_2O
MgO
$NiCoFe_2O_4$
Dissolve with distilled water
Mixing all three solutions in 500ml beaker
Stirring and Heating (15 min)
Add 0.1 g of biochar
Stirring for 20 min
Add leaf extract
Heat and stir 4-6 hours
Filter dry 24 hour (85°C) and store it dry glass bile.

Scheme 2: Synthesis of biochar supported Ag-Mg nanocomposite encapsulated nickel-cobalt-ferrites.

5.4. Synthesis of Nickel-cobalt-ferrites

To synthesize Nickel doped Cobalt Ferrites, a solution of $FeCl_2 \cdot 4H_2O$ (0.4 M) and $NiSO_4 \cdot 6H_2O$ (0.1 M) was prepared in 50 mL of distilled water, and $CoCl_2 \cdot 6H_2O$ (0.3 M) was dissolved in 25 mL of distilled water with suitable heating to dissolve the salts. The Nickel doped Cobalt Ferrites mixture was prepared by adding the $NiSO_4.6H_2O$ (0.1M) and $CoCl_2.6H_2O$ (0.3M) solutions to the $FeCl_2.4H_2O$ (0.4M) solution with continuously stirring and heating at 40^0C. Added NaOH (3.0M) solution dropwise to this solution to maintains pH value 10-11 with constant stirring at 80^0C for 1 hour till the precipitate formed. Filtered the precipitate using whatman filter paper. Dried the precipitate by heating in an oven at temperature 90-100^0C for 24 hrs and stored in a dry glass vile.

5.5. Characterization Techniques

5.5.1. Fourier transforms infrared (FTIR) analysis

Fourier transform infrared spectroscopy is a technique which provides sufficient information about the structure of organic compounds. It helps in the identification of unknown compound, determine the quality of a sample and quantity of components present in the mixture. This technique based on the principle that when the IR radiation is passed through a sample, some of radiations are absorbed or transmitted by the sample. Radiations absorbed by the sample caused the excitation of molecules from lower to higher vibrations.

5.5.2. X-ray diffraction (XRD) pattern

X-ray diffraction (XRD) pattern is a powerful, non-destructive and rapid technique. It is widely used for the identification of crystal structure of an unknown material and determination of sample purity. The analyzed material is commonly present in the powder form or finely grounded.

5.5.3. Field emission scanning electron microscope (FESEM)

FESEM instrument is widely used for determining the morphological anatomy of an object. Field emission scanning electron microscope (FESEM) scans the surface of an object with high energy beam of electrons. These electrons interact with atoms to produces the signals. This signal contains secondary and back scattered electrons that produces microscopic FESEM images or used for surface imaging.

5.5.4. High resolution transmission electron microscope (HRTEM)

High resolution transmission electron microscope (HRTEM) is an important tool that provides the necessary information such as crystal structure, internal morphology, compositions, particle size etc. HRTEM images were captured on a FP5022/22-Tecnai G2 20S-TWIN (USA) transmission electron microscope at acceleration voltage of 200 kV.

5.5.5. Energy dispersive X-ray (EDX)

Energy dispersive X-ray spectroscopy (EDX) is another powerful non-destructive technique used to determine the elemental compositions. The electron beam excites the atoms in the sample and consequently, produces the X-rays to discharge the excess energy. EDX spectra are recorded from a particular point on the sample. Alternatively, electron beam sweeps over a selected area of a sample to identify the presence of elements.

5.5.6. BET Surface Area Analysis

This is a method used to measure the specific surface area of materials. The theory extends the Langmuir theory of adsorption to multilayer adsorption. A sample is degassed to remove any moisture or contaminants. Then, nitrogen gas is introduced, and the amount of gas adsorbed at different pressures is measured. The BET equation is then used to calculate the specific surface area of the material. This method is crucial in characterizing materials like catalysts, porous materials, and powders.

5.5.7. Vibrating Sample Magnetometry (VSM)

VSM is a technique used to measure the magnetic properties of materials. In VSM, a sample is placed in a magnetic field and is vibrated at a specific frequency. The vibration causes the magnetic dipoles within the sample to oscillate. The oscillating magnetic dipoles induce an alternating voltage in a nearby pickup coil. This voltage is proportional to the magnetization of the sample. The sample is subjected to a magnetic field, which can be varied during the measurement to study how the material responds to different magnetic fields.

5.6. Photocatalytic degradation

The photocatalytic degradation of biosynthesized NP's was evaluated by the pollutant degradation under sunlight irradiation. Then 1.0 mg of photocatalyst was added in 100 mL (1×10^{-5} M) of pollutant aqueous solution was stirred in the dark condition for 10 min until equilibrium condition attains followed by continuous stirring. Afterwards, suspension was kept under sunlight and readings were taken for each 10 min up to 50 min. The suspension was changed from X colour to colourless. The final solution was analysed by using UV-vis spectrophotometer. The percentage of pollutant absorbed on the catalyst surface was calculated by following equation:

$$\text{Photocatalytic degradation}(\%) = \frac{A_0 - A_t}{A_0} \times 100\% \quad (1)$$

Where A_0 is initial in absorption and A_t is absorption after variation.

5.7. Adsorption Experiment

The effect of initial adsorbate concentration and time was evaluated in this experiment. Adsorbent (0.1 g) was added to solutions (10 mL) of pollutants at different concentrations. All adsorption experiments were done at 30 °C using an orbital shaker operating at 200 rpm. The residual concentrations of pollutants were analyzed after predetermined intervals of time until the system reached equilibrium. The pollutant adsorbed at equilibrium (mg/g) was calculated using the following equation:

$$q_e = (C_i - Ce)\,\frac{V}{M} \quad (2)$$

Where, C_i and Ce indicates initial and equilibrium concentrations (mg/L), respectively. q_e (mg/g) express equilibrium adsorption capacity, V corresponds to volume of solution (ml) and M is mass of nanocomposites (mg/L). The percentage adsorption of pollutant was calculated as:

$$\%\ \text{Removal} = \frac{C_i - C_e}{C_i} \times 100 \quad (3)$$

C_i and C_e (mg/L) are initial and final pollutant concentrations before and after adsorption.

5.8. Adsorption isotherms

Adsorption isotherms are essential for understanding the interaction mechanisms between adsorbates and adsorbents and for evaluating adsorbent performance. These isotherms depict the ratio between the quantity of adsorbate adsorbed and its equilibrium concentration in the aqueous phase, illustrating the relationship between pollutant concentration and uptake. Various isotherms are used to analyze adsorption process data. In this study, the experimental data were interpreted using three prominent models: Langmuir, Freundlich, and Tempkin isotherms.

5.8.1. Langmuir isotherms

The Langmuir isotherm is expressed by the following equation:

$$q_e = \frac{K_L q_m C_e}{1 + K_L C_e} \quad (4)$$

where q_e is the amount of adsorbate adsorbed at equilibrium in mg g^{-1}, q_m is the maximum monolayer adsorption capacity of the adsorbent in mg g^{-1}, C_e is the concentration of the adsorbate under equilibrium conditions in mg L^{-1} and K_L is the Langmuir constant in L mg^{-1}. The Langmuir isotherm can also be expressed in the following equation to determine the constants K_L and q_m:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{K_L q_m C_e} \quad (5)$$

The values of the constants can be found from the intercept and slope of the linear plots of the experimental data of ($1/q_e$) versus ($1/C_e$) for the different dyes.

5.8.2. Freundlich isotherms

The Freundlich isotherm is expressed by the following equation:

$$q_e = K_F C_e^{\frac{1}{n}} \quad (6)$$

Where K_F and n are Freundlich constants. K_F is the adsorption capacity of the adsorbent in mg g^{-1} (L mg^{-1}) and it represents the amount of adsorbate adsorbed onto the adsorbent for a unit equilibrium concentration. The values of the constants can be determined from the intercept and slope of the linear plots of the experimental data of (ln q_e) versus (ln C_e) for the different dyes and metal ions, following the equation below:

$$\ln q_e = \ln K_F + \frac{1}{n}\ln C_e \quad (7)$$

5.8.3. Tempkin isotherms

Tempkin and Pyzhev isotherm model may be presented by following equation:

$$q_e = \frac{RT}{b}\, ln(K_F\, C_e) \quad (8)$$

Linearized Tempkin isotherm equation can be written as follow:

$$q_e = B_i \ln(K_T) + B_i \ln (C_e) \quad (9)$$

$$B_i = \frac{R_T}{b} \quad (10)$$

where, T is absolute temperature (K), R is universal gas constant (8.314 J mol^{-1} K^{-1}) and fitting plot between q_e and ln C_e determine the values of B_i and K_T, which were computed from the slope and intercept, respectively.

5.9. Kinetic study

Adsorption is a time dependent mechanism and inspected in terms of many processes such as diffusion model and chemical reaction. The linear equation for pseudo-first-order can be written as:

$$\log(q_e - q_t) = \log q_e - \frac{K_L}{2.303} \times t \quad (11)$$

Where, q_e and q_t are the adsorption capacity at equilibrium and at time t, respectively.

5.10. Thermodynamic study

The nature of adsorption process will be explained in terms of thermodynamic parameters such as Gibbs free energy (ΔG^O), enthalpy (ΔH^O) and change in entropy (ΔS^O). The equation for thermodynamics can be represented as:

$$\Delta G^0 = \Delta H^0 - \Delta S^0 \quad (12)$$

In order to investigate whether the adsorption is spontaneous and explore the mechanism involved in the adsorption process the thermodynamic parameters can be determined by following equations:

$$\Delta G = -R_T \ln K_d \quad (13)$$

$$lnK_d = \frac{\Delta S}{R} - \frac{\Delta H}{R_T} \quad (14)$$

where, ΔS (J $mol^{-1}k^{-1}$), ΔH (J $mol^{-1}k^{-1}$) and ΔG (J $mol^{-1}k^{-1}$) are the entropy change, enthalpy change and Gibbs free energy change respectively. R (8.314 J mol^{-i} k^{-1}) is the gas constant and T (K) is the temperature.

6. SIGNIFICANCE

This study's effectiveness to others and the environment lies in its potential to contribute valuable insight or innovations to enhance adsorption-based pollutant removal process. By conducting research in this field, we can optimize the adsorbent materials, modify operating conditions or to develop novel techniques. This not only benefits wastewater treatment but also reduce the release of harmful pollutants into natural eco-system, safeguarding the human health and environment system. It also plays a crucial role in advancing sustainable and eco-friendly solution from wastewater treatments, which are increasingly vital in today's world facing water scarcity and pollution challenges. In recent years, the contamination of water systems with persistent pollutants has emerged as a critical environmental issue. Persistent pollutants, such as heavy metals, organic contaminants, and industrial chemicals, pose severe risks to ecosystems and human health. Traditional water treatment methods often struggle to effectively remove these contaminants due to their complex nature and low concentrations. In this context, advanced materials and technologies are being explored to enhance the efficiency of water remediation processes. One promising approach involves the use of activated biochar-supported nano-hetero assemblies. This combination leverages the unique properties of activated biochar and nanotechnology to address the challenge of persistent pollutants in contaminated water systems. This essay explores the significance of this innovative approach, provides examples, and discusses its potential impact on water remediation. The significance for the removal of pollutants by adsorption technique lies in its effectiveness in improving water quality and mitigating environmental pollution. Adsorption offers as a versatile and efficient method for the removal of pollutants and contaminants on broader scale.

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COMMENTS FROM EXTRNAL EXPERT

The following suggestions has been given by an external subject expert:

1. **Comment** – Anti-cancer drug not to be prefer as it could be exposure to toxic substances which could lead to adverse reaction or long-term health issues.

   Response – Based on the suggestion provided by the senior expert, anti-diabetic agents, pesticides, or herbicides will be in our research applications.
2. **Comment** – Botanical name of plant should be italic with first letter is uppercase.

   Response – The changes have been made in accordance with the expert's suggestion.
3. **Comment** – Thermogravimetric analysis (TGA) to confirm the biochar formation temperature as it measures changes in mass as temperature increases.

   Response – As suggested, Thermogravimetric Analysis (TGA) has been utilized to confirm the biochar formation temperature, given its capability to track mass changes with temperature increases.
4. **Comment** – In place of term "Bio-nanocomposite" use "Nanocomposite" at higher temperature as it used for composites consists biochar and at higher temperature biochar presence in composite must be affected due to thermal degradation.

   Response – Based on the suggestion, we have adopted the term "Nanocomposite" in place of "Bio-nanocomposite," recognizing that biochar may degrade at higher temperatures, influencing its role in the composite.